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DILATOMETRIC INVESTIGATION OF THE GROWTH OF CRYSTALS AT HIGH TEMPERATURES

Ya. I. Ol'shanskiy
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Presented by Acad D. S. Belyankin
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/Figures are appended./

Certain results of the investigation of the growth of crystals when solid monophase colloids are heated at high temperatures have already been reported in a preceding work by the author (Doklady Akademii Nauk SSSR, Novaya Seriya, Vol LXII, p 489, 1948). It seemed expedient to study this process also by other methods. Therefore, we settled on dilatometry, which makes it possible to observe directly the rate of the process in relation to the temperature and the duration of heating. Moreover, such measurements permit certain observations to be made about the variation of the coefficient of expansion of monophase colloids in dependence on their degree of dispersion. The problem is also of interest from the viewpoint of experimental technique, since dilatometric analysis at temperatures exceeding 2000° has not been used hitherto as far as we know.

Samples of petroleum and fused coke, from which slabs of 1 x 1 x 5 cm were sawed, were taken as the subject for investigation. A diagram of the apparatus is shown in Figure 1. The slab (1) is placed on the graphite block (2), which simultaneously serves as a standard for comparison of the coefficients of expansion, and is inserted in the center of the heated carbon furnace tube (7).

The elongation of the sample relative to the standard is measured by means of the mirror (3) and a tube calibrated for measurement. For transmission of the elongation, two carbon rods (4) are used, one of which is connected with the hinge of the mirror, and the other with a conical point which tilts the mirror when the expansions of the sample and the standard are different.

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The constant tension of the carbon rods is maintained by two identical small weights with the aid of pulleys and string (not shown in the figure). To avoid significant buckling, the carbon rods pass through a series of graphite disk-shaped washers, which also reduce radiation into the cold part of the furnace tube.

From the other side, the block (2) is supported by a carbon rod which, like the mirror mechanism, is fastened to the metal frame.

To decrease the natural movement of the apparatus, the carbon rods were heat treated under identical conditions (simultaneously) at 2,800° for 2 hr. To a significant degree, this equalized a certain difference of their coefficients of expansion which was always observed. Temperatures were measured with an optical pyrometer by sighting through the aperture (5) in the block. During temperature measurements nitrogen was blown through the furnace tube entering through the glass tube (6) provided with a plane-parallel glass port. (Note: At high temperatures the appearance of smoke is always observed in carbon furnaces, and the measurement of temperature with an optical pyrometer without blowing can lead to a very large error.)

To determine the natural movement of the device, the test slab sawed from samples of coke was replaced by another slab sawed from the same graphite as the block. The natural movement of the apparatus was quite constant up to 2500° and did not exceed 5% of the movement occurring in work with the samples being tested. Measurements at higher temperatures were impossible, as the carbon rods begin to be noticeably deformed.

Measurements of the elongation or contraction of the samples relative to the standard were made in a range extending through 100-2000°. The temperature was held at each point until the mirror of the device stopped moving, and for this 30-60 minutes were necessary at low temperatures, while 10-15 minutes were sufficient at high temperatures (above 200°).

In Figure 2 are shown the results of the dilatometric measurements of two samples of petroleum coke and one sample of fused coke which were first enriched at 1,200° for 3 hr. The behavior of all the samples was very consistent. The lower branch of the curve refers to the process of heating to the temperature of preroasting. The coefficient of thermal expansion of the samples studied is greater than that of the standard (graphite), and the difference between them grows with the temperature.

After the temperature was raised above 1,200°, the samples were observed to contract, and this continued up to the highest temperatures. This part of the curve is irreversible, and it refers to the process of crystal growth. The upper branch corresponds to the contraction of the samples, which is subjected to heating up to 2,425°. The coefficient of thermal expansion in the temperature interval of 200-1,000° becomes less than for the initial substance, but remains greater than for the standard.

Numerous observations with the aid of the dilatometer permitted the drawing up of a systematic graph (Figure 3), which qualitatively characterizes the process of enlargement of the crystals on heating. Curves I, II, III, IV, V, and VI give a representation of the thermal expansion of substances for different temperatures of preheating: 900, 1,580, 2,080, 2,450, and 2,380°, respectively.

Curve VII refers to the process of irreversible contraction with the enlargement of the crystals of the monophase colloid. For example, if we take a sample of petroleum coke which has been subjected to prior heating at 1,200° and observe its expansion with heating, then its thermal expansion up to 1,200° is shown by Curve II, which can also be reproduced for the cooling process.

On heating higher than 1,200° an irreversible contraction begins, accompanying the enlargement of the crystals. If the heating is terminated at 1,580° and cooling begun, then the sample being tested begins to contract according to Curve III. The coefficient of linear expansion under the circumstances is less than for the initial substance.

On the second heating of the same sample, the thermal expansion up to 1,580° is shown by Curve III, but a further increase of the temperature is accompanied by irreversible contraction (Curve VII), since from this temperature the growth of the crystals is resumed. The smooth path of Curve VII shows that the process of growth of the crystals proceeds at all temperatures without abrupt jumps. To each temperature (when heating is not of excessive duration), there corresponds a certain average size for the crystals. The decrease of the coefficient of thermal expansion with the enlargement of the crystals seems to be characteristic.

[Appended figures follow.]

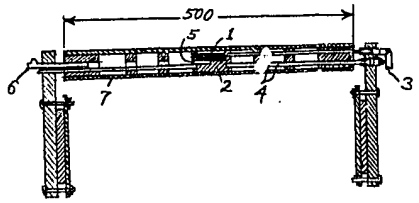

Figure 1. Apparatus Used for the Investigation:

1. slab; 2. block; 3. mirror; 4. carbon rods; 5. aperture in block; 6. glass tube; 7. carbon furnace tube

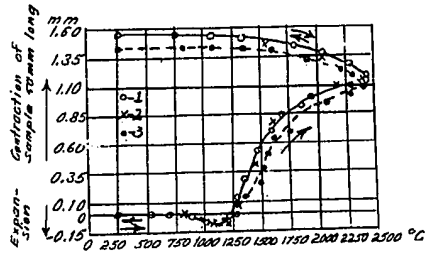

Figure 2. Measurements of Petroleum Coke and Fused Coke

1, 2. petroleum coke; 3. fused coke

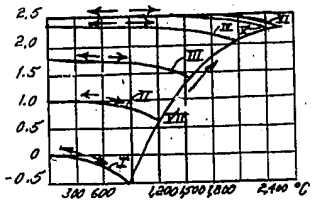

Figure 3. Crystal Growth as Influenced by Heating

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